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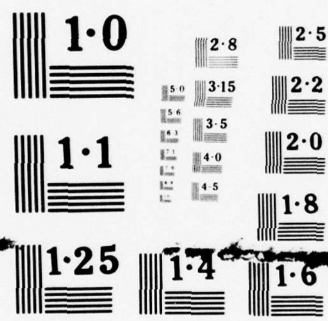

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DEFENCE RESEARCH ESTABLISHMENT OTTAWA

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APPLICATION OF ULTRASOUND AND MICROWAVES DURING TREATMENT OF FABRICS WITH LIQUID-REPELLENT FINISHES

by

J.M. McAndless, B.V. Lacroix, A.J. Last and T.W. Hislop

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ABSTRACT

Two types of military fabric were treated with a liquid-repellent fluorochemical finish in the presence of high-frequency sonic energy (ultrasound) over a range of frequencies and power levels. Fabric properties such as physical strength, liquid repellency, and durability of repellency to laundering and wearing were evaluated following treatment to determine the effects of using ultrasound during the finishing process.

A second series of tests was carried out to determine whether microwave energy could be employed for the purpose of ensuring a more uniform impregnation of the fabrics, as well as to accelerate the drying and curing processes.

RESUME

Deux sortes de tissus utilisés dans les forces armées ont étés traités avec un fini fluoro-chimique ayant des propriétées hydrofuges en présence d'ultrasons (énergie sonique à haute fréquence) selon différente fréquence et intensite de pulsation. On a évalué les propriétées du tissu traité telle que, la solidité des tissus, la résistance à l'huile et à l'eau, la durabilité de ces propriétées à l'usure et au lavage pour déterminer les effects de l'emploi des ultrasons lors de l'application du fini.

Une deuxième série d'essais ont été fait dans le but de savoir si les micro-ondes pourraient être utilisées afin d'assurer une imprégnation plus uniforme du materiel aussi bien qu'accélérer le séchage et la polymerisation du fini.

INTRODUCTION

Certain fluorinated polymers, unique in their ability to impart both oil- and water-repellency to textiles while leaving the fabric permeable to air and water vapour, are used extensively as liquid-repellent finishes for protective clothing. However, these so-called "fluorochemicals" are extremely expensive; so for economy reasons and sometimes to bring about improvements in water repellency, fluorochemicals are often extended with conventional water-repellent compounds in finishing formulations.

Prior work (1, 2) has shown that fabrics treated with conventional fluorochemical/water-repellent finishes tend to rapidly lose oil repellency under certain laundering and wearing conditions to the point where the protection provided is not adequate for military purposes. This problem can be partly overcome by treating fabrics with fluorochemical alone or with fluorochemical/resin finishes in which the resin extender does not contain the type of long-chain hydrocarbon substituent usually found in water repellent compounds (2, 3).

In a preliminary investigation into new methods which might provide for more effective utilization of fluorochemical finishes, the application of high-frequency sonic energy (ultrasound) during treatment of fabrics with an aqueous fluorochemical solution was found to produce an increase in finish add-on over the standard treatment method (4). This study was limited by the fixed frequencies and low power output of the ultrasonic equipment used to insonate fabric samples during the finishing process.

Following the preliminary study, a comprehensive examination of the application of ultrasound during the treatment of fabrics with fluorochemicals was undertaken to determine whether any beneficial effects such as increased finish add-on could be optimized. As described in this report, two types of military fabric were treated with a commercial fluorochemical in the presence of specially designed ultrasonic equipment capable of operating at 8.7, 22.8, 46.6 or 860 kHz and at different power levels. Fabric samples were also treated using conventional methods and the various samples were compared with respect to their physical properties, liquid repellency and durability of liquid repellency to laundering and wearing.

Literature searches revealed that the use of microwave heating during post-treatment drying of textiles results in an improved uniform deposition of dyes and other finishes such as flame retardants when compared to conventional surface heating (5, 6). As part of the present study, an investigation into the effects of microwave drying on the properties of fabrics treated with the fluorochemical finish was carried out. Freshly treated fabric samples were air dried and/or dried by passing through a specially designed microwave guide operated at a power output of 1500 watts and then cured in the conventional manner. Comparison tests between these fabrics and those dried and cured under the same conditions without microwaves were carried out.

EXPERIMENTAL

CHEMICALS

Fluorochemical FC-232[a] was used as the liquid-repellent finish during this study. This finish is a water-based fluoropolymer, supplied by 3M Company as an emulsion of 30% solids content by weight.

FABRICS

Two fabrics (Table I) were chosen for treatment with the fluorochemical finish. These fabrics were representative of the types of light-weight and heavy-weight fabrics used in military clothing and equipment systems.

[a] Use of a commercial product does not imply recommendation or approval of that product by the Department of National Defence to the exclusion of other products.

TABLE I

Fabrics Used in Finishing Studies

Designation	Fabric	Description
NC-5	Nylon/Cotton	50/50 twist blend, OG107 dye, 170 g m^{-2} (5 oz yd^{-2})
PC-8	Polyester/Cotton	65/35 twist blend, OD7 dye, 282 g m^{-2} (8.3 oz yd^{-2})

FABRIC FINISHING

Treatment Solution

A solution containing 10% by weight (3% solids) FC-232 emulsion in tap water at room temperature was used for finishing fabrics. This solution was stable but if it had stood three weeks or more a fresh solution was made up prior to fabric treatment.

Treatment Cycle

Fabric samples of 20 cm x 40 cm size were put through the following standard treatment cycle:

1. Triplicate samples were weighed dry to 0.01 g accuracy and then passed through the treatment solution at a velocity of 2.5 cm per second;

2. The treated fabrics were then passed through an Atlas Laboratory Padder, Model LW-1, with the rolls set at 27.2 kg (60 lbs) pressure to remove excess solution;

3. In some cases, fabrics were passed through the treatment bath and the padder rollers a second time (two-dip/two-nip treatment);

4. Damp samples were weighed as rapidly as possible before any appreciable air drying took place;

5. Weighed, damp samples were hung up on cotton strings to air dry overnight;

6. Air-dried samples were weighed to the nearest 0.01 g and then cured in a laboratory oven at 170°C for 2 minutes on special racks;

7. Samples were then cooled to room temperature and reweighed.

Treatment Cycle Using Ultrasound

Samples of 20 cm x 40 cm size were put through the standard treatment cycle as described above with the following variations:

1. Tx-Contact: fabrics were pulled through the treating solution while rubbing against the working face of the immersed "blade" of an ultrasonic transducer.

2. Tx-Remote: the fabric was kept at least 1 cm away from the working face of the immersed transducer blade as the sample was passed through the treatment bath.

3. Both Tx-Contact and Tx-Remote tests were run with ultrasound or with no ultrasound to give four treatment combinations at a single frequency and power level.

4. Fabrics were insonated at frequencies of 8.69 kHz, 22.80 kHz, 46.60 kHz and 860 kHz.

5. Ultrasonic power levels were chosen to include high power (over 100 watts net) in the cavitation range and moderate power (approximately 15 watts net) below the cavitation range.

6. The 860 kHz transducer was operated at its normal power level (approximately 50 watts) as the forces in this frequency range favour acceleration rather than cavitation. The method of treating fabrics while using this transducer was modified to accomodate the relatively narrow beam of ultrasonic energy produced (see Equipment).

Treatment Cycle Using Microwaves

Leader strips (140 cm) were attached to both ends of 20 cm x 215 cm fabric samples with adhesive tape. This combined length was

sufficient to conduct the treatment cycle using the padder rollers as a transport, operating at 2.5 cm per second linear speed. The strips were first drawn through the fluorochemical bath (Tx-Contact mode) and padder rollers as in the standard single-dip/single-nip treatment cycle. The damp strips were then drawn through slots in the microwave 3-pass serpentine wave guide (see Equipment). It was found that complete drying by microwave necessitated more than one pass through the wave guide. Four passes in the case of the nylon/cotton fabric and six passes for the polyester/cotton fabric were considered adequate. In some cases, samples were air dried following microwave treatment. All samples were then cured at 170°C for 2 minutes with the exception of one polyester/cotton sample which had gone through the wave guide six times.

EQUIPMENT

Ultrasonic Equipment

Figures 1 and 2 show the 8.7-kHz and 22.8/46.6-kHz transducers respectively which were designed and built for use in this study. These transducers were driven at the indicated frequencies by a Macrosonics Corporation Model KC 500-1 Multifrequency Generator with power levels monitored by a Wave Energy Systems Wattmeter, Model M1/SC1. The general layout of the equipment and the operation of the appropriate transducer in the Tx-Contact mode is illustrated in Figures 3a and 3b.

Figure 4 shows the transducer which was used for tests carried out at 860 kHz. For these tests, a treatment tank 29 cm long x 23.5 cm wide x 15.2 cm deep was used to contain 5 liters of 10% FC-232 solution. The transducer (Tx) was fixed to the bottom of the tank with masking tape, with its ultrasonic beam of about 5 cm diameter working upwards as indicated in Figure 5. The transducer was driven by a Macrosonics Corporation aerosol Generator, Model 250 FF. To ensure complete insonation over the entire area of the fabric samples, they were drawn manually back and forth through the treating solution, making 7 or 8 passes as indicated in the Figure. This process required 3 minutes to complete; therefore, control samples (not insonated) were soaked for the same length of time. Following treatment in this manner, samples were passed through the padder rollers, air-dried and cured as before.

Microwave Equipment

The set-up for the microwave drying tests is illustrated in Figure 6. The microwave generator employed was a Microwave Technology Model 2212 Power Unit, producing 2,450 MHz microwaves with variable

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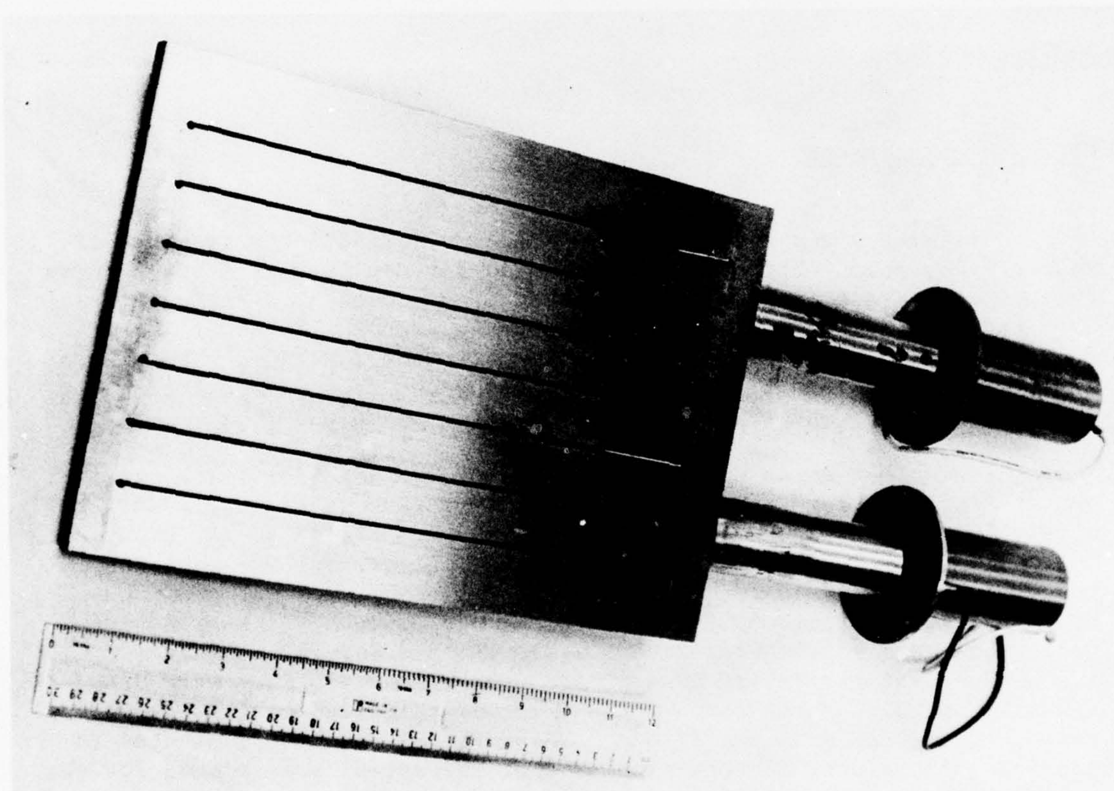

Figure 1. Ultrasonic Transducer for 8.7 kHz Operation

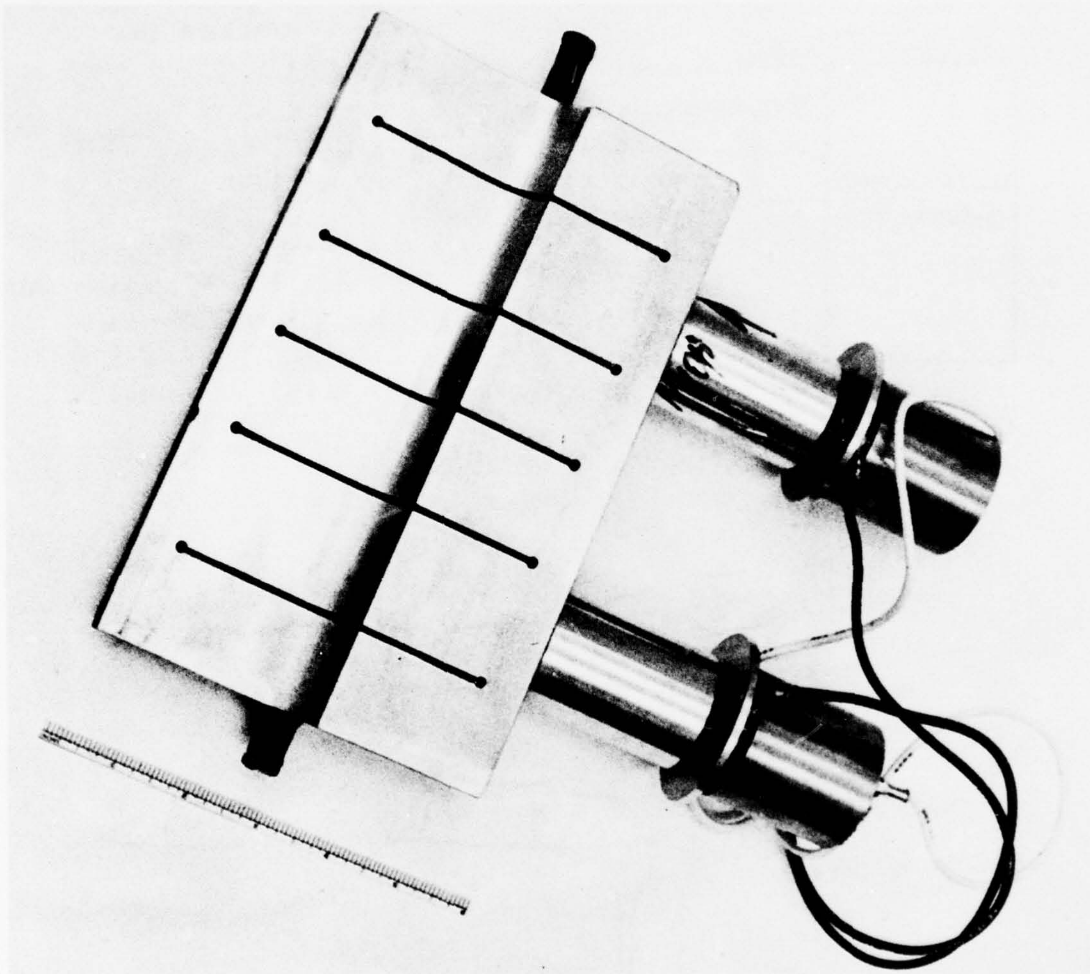

Figure 2. Ultrasonic Transducer for 22.8- and 46.6-kHz Operation

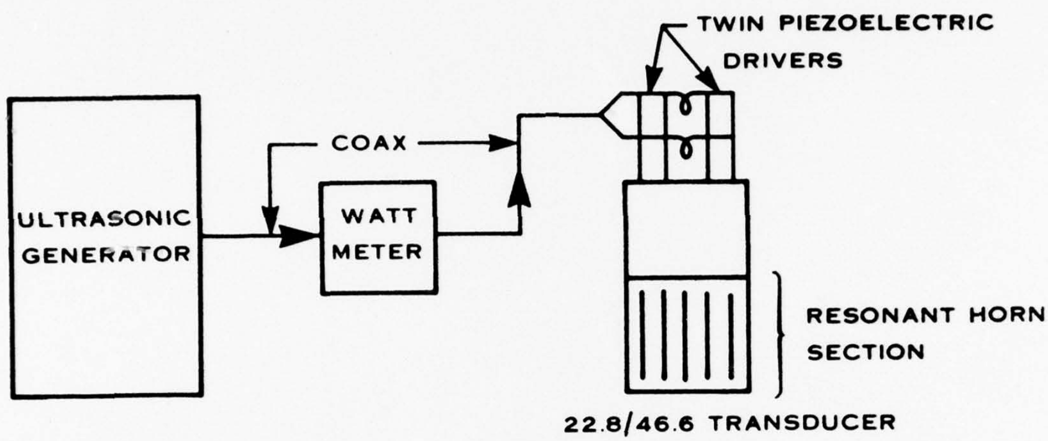

Figure 3a. General Arrangement of Ultrasonic Equipment.

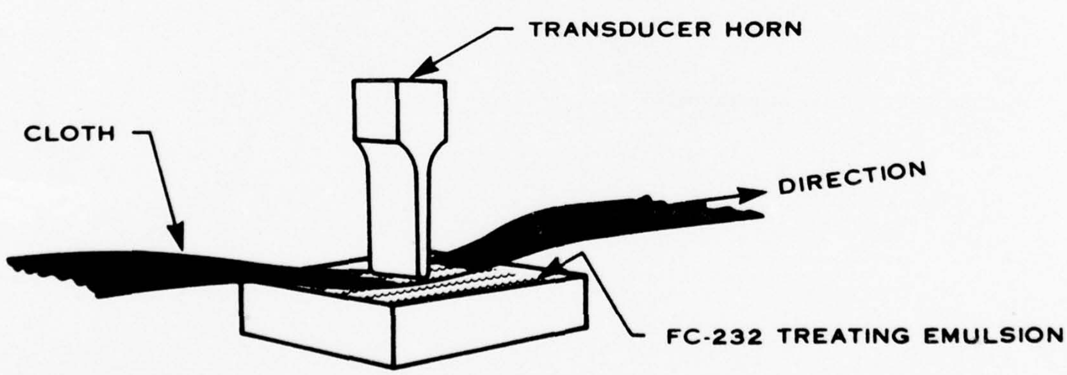

Figure 3b. Tx Contact Tests.

Figure 4. Ultrasonic Transducer for 860 kHz Operation

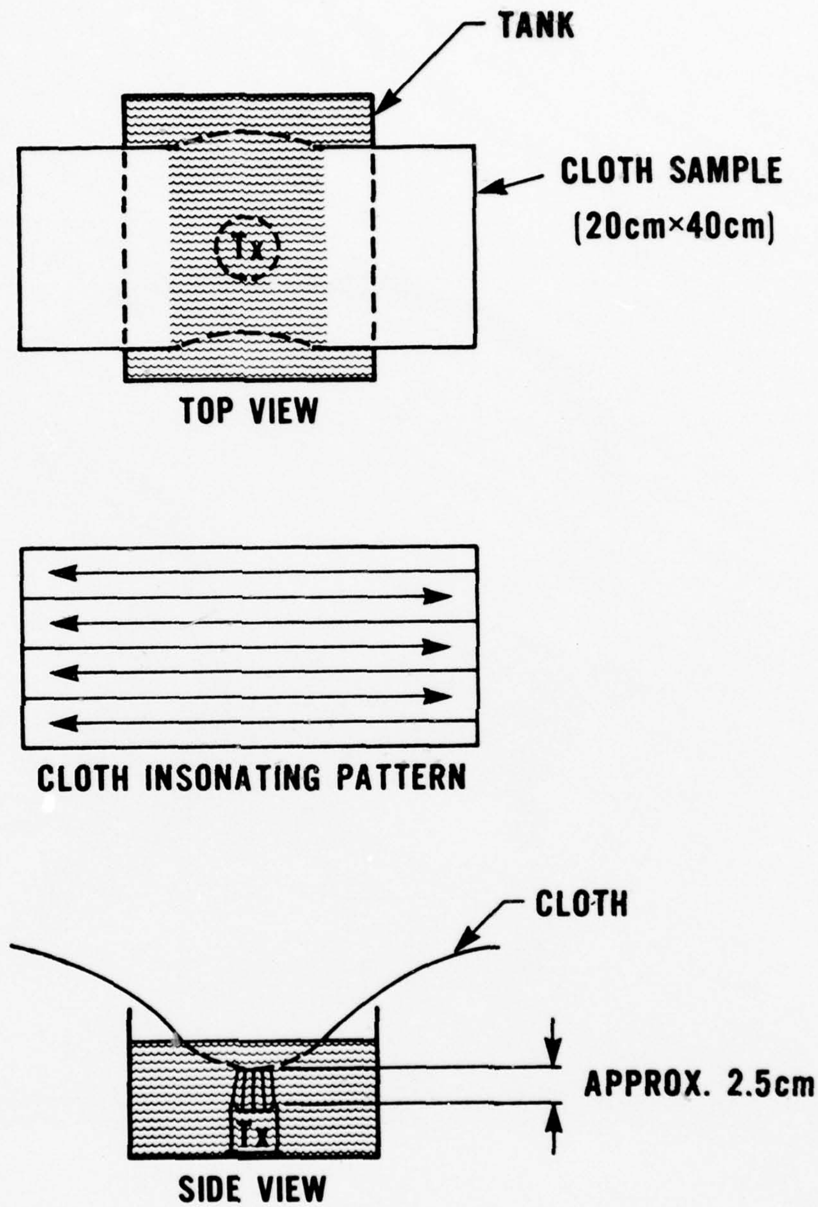

Figure 5. Arrangement for Insonating Fabrics Using The 860 kHz Transducer.

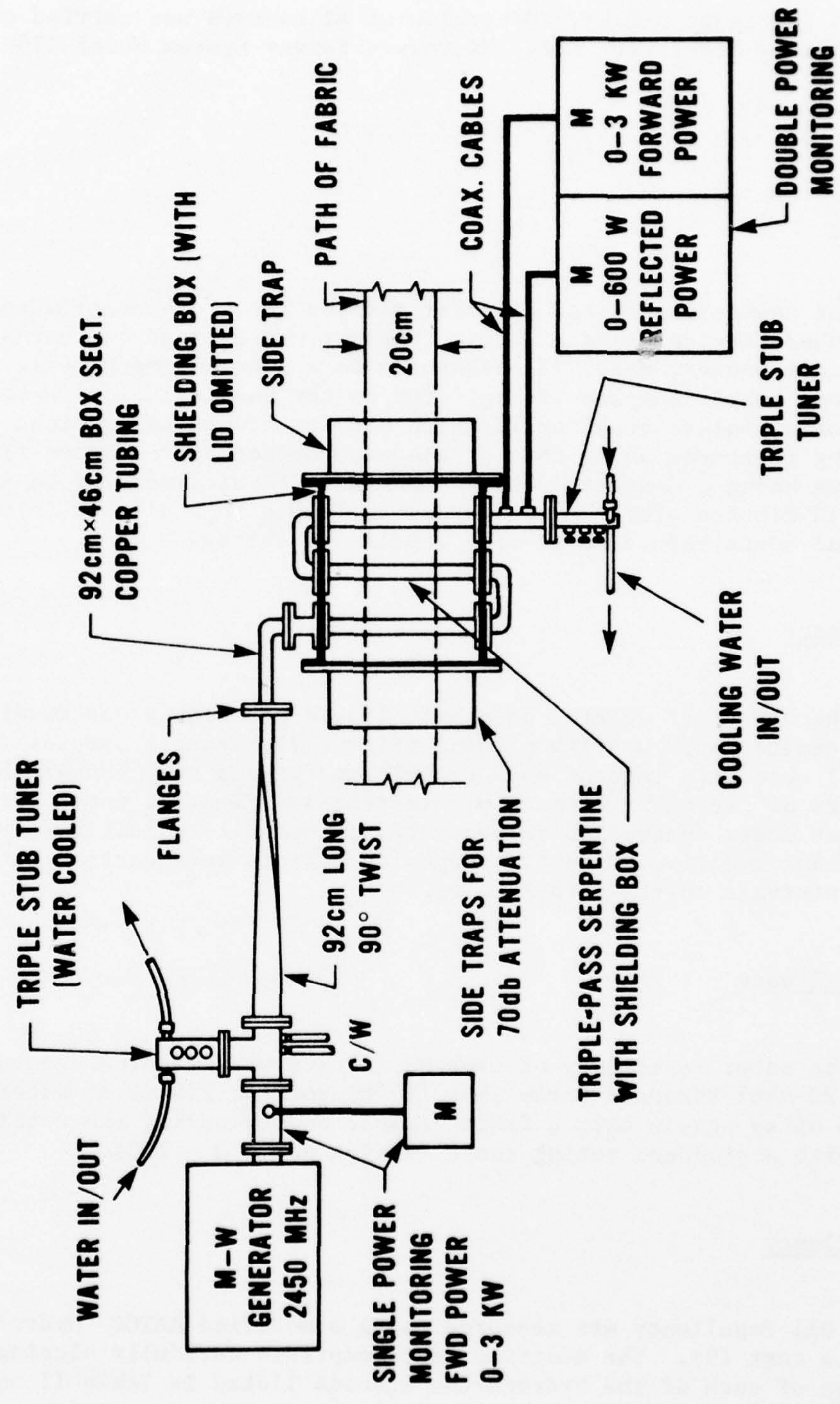

Figure 6. Arrangement of Microwave Equipment.

power over the range 0-2 kW. Detection of microwaves was carried out using a Holiday Industries Ltd. Microwave Survey System Model 1700.

TEST METHODS

Laundering

For laundering tests, circular samples (8 cm diameter) were cut from finished fabrics using a punch. Washing was carried out using an Atlas Launder-Ometer, Model B5, according to a standard method(7). In this method, fabric samples are agitated in hot soap solution in the presence of stainless steel balls which provide mechanical action. One washing treatment using this accelerated method approximates five launderings using a domestic washing machine. Fabrics were dried at 90°C for 10 minutes after each cleaning cycle and then allowed to stand for several hours before commencing repellency tests.

Wearing Tests

The effect of wearing on fabric liquid repellency was examined using an experimental wearing machine (Figure 7). Fabric samples (18 cm x 27.5 cm) were sewn into an endless belt and passed over the brushes and rollers of the machine under 0.5 kg tension. Wearing tests were carried out under controlled temperature and humidity conditions; viz. 22°C and 55% relative humidity. Repellency tests were carried out at regular intervals on the worn fabrics.

Water Repellency

The water repellency of treated fabrics was measured according to AATCC 22-1967 Standard Spray Test (8) by pouring 250 ml of water through a spray nozzle onto a fabric sample and comparing the wetting pattern with a standard rating chart (Rating scale 0 - 100).

Oil Repellency

Oil repellency was measured using a modified AATCC hydrocarbon-resistance test (9). The modified test comprises carefully placing a small drop of each of the hydrocarbon liquids listed in Table II on the

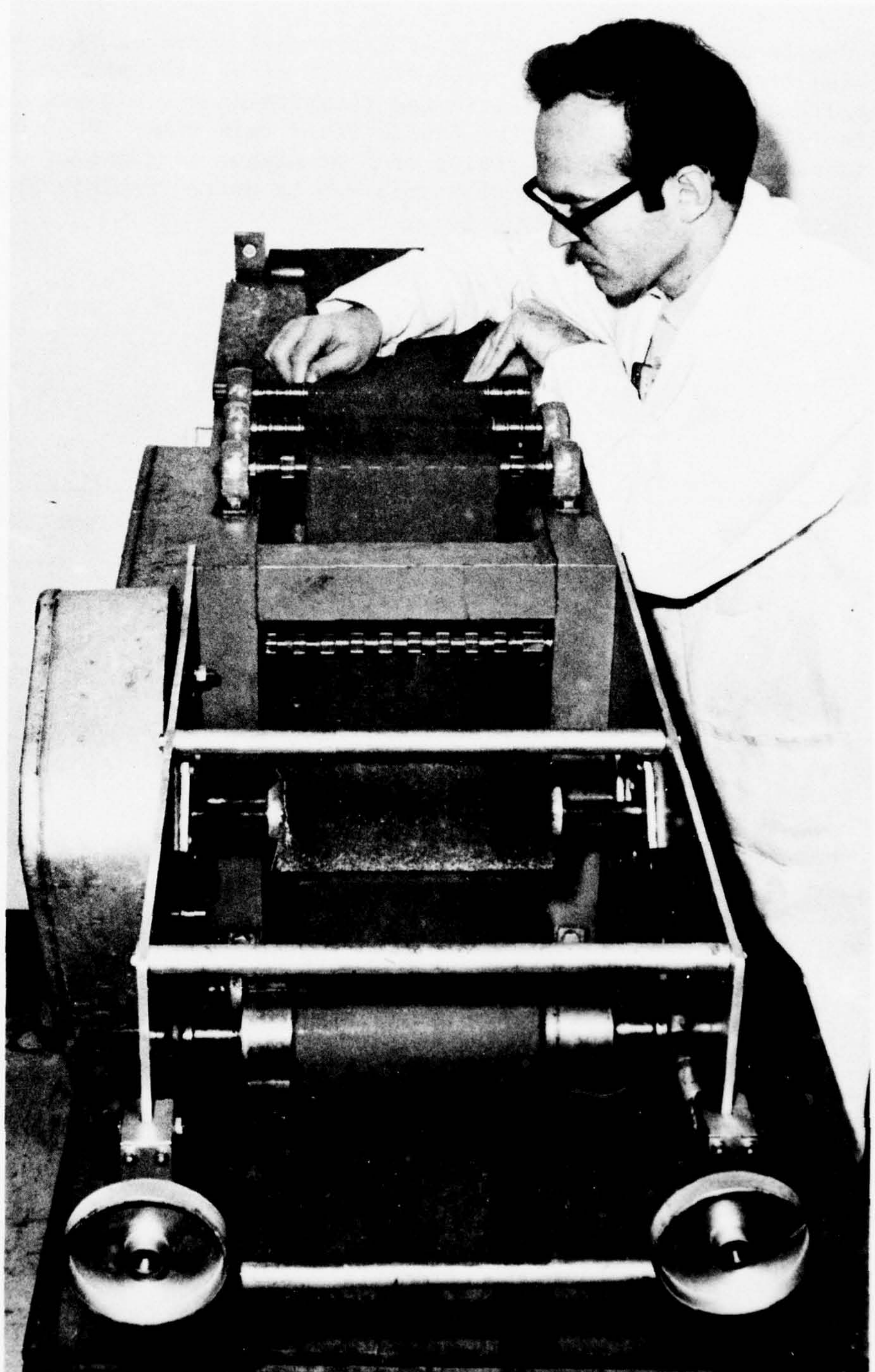

Figure 7. Wear-Testing Machine

fabric sample which is lying on a flat horizontal surface. Any penetration or wicking into the fabric was noted visually after five minutes. The oil repellency rating of the fabric was recorded as the highest-numbered test liquid which did not wet the fabric after this time. With this test, (rating scale 0 - 7), a rating of 5 or higher is considered good; a fabric with a rating of less than this can be wetted rapidly by most common fuels and low-viscosity oils.

TABLE II

Oil-Repellency Test Liquids

Rating Number	Hydrocarbon Liquid	Proportions
1	Nujol	
2	Nujol/n-hexadecane	65/35
3	n-hexadecane	
3/4	n-hexadecane/n-tetradecane	50/50
4	n-tetradecane	
4/5	n-tetradecane/n-dodecane	50/50
5	n-dodecane	
5/6	n-dodecane/n-decane	50/50
6	n-decane	
6/7	n-decane/n-octane	50/50
7	n-octane	

Phosphate Resistance

The resistance of treated fabrics to wetting by organo-phosphorus liquids was determined in a manner similar to the oil-repellency test. Small drops of the model test liquids trimethyl phosphate, triethyl phosphate and tri-n-propyl phosphate were placed on a flat fabric sample. After one hour, the appearance of each drop was noted visually and a rating assigned to the fabric based on the overall appearance of the three types of droplets (rating scale 0 - 9). A rating of 7 or above is considered

good and means at least two of the three phosphate liquids have shown no signs of wetting or penetrating into the fabric. A rating of less than 4 indicates the fabric has been wetted to some extent by all three of the liquids.

Fabric Strength

The physical properties of representative fabric samples were determined before and after treatment under the various conditions using standard tearing tests (10) and breaking strength tests (11).

RESULTS

FABRIC FINISHING USING ULTRASOUND

Three identical samples of both the light- and heavy-weight military fabrics were treated with fluorochemical for each of the four types of treatment cycles (Tx-Remote, Tx-Contact, U/S[b]-Tx-Remote, U/S-Tx-Contact) described previously. The Tx-Remote cycle, since it involves neither contact between the fabric and transducer nor use of ultrasound, is analogous to the standard method of fabric treatment. Individual samples were numbered and each group of three fabrics run through a given treatment cycle was designated as a series (A, B, C, etc.) to facilitate comparison of results.

Finish Add-On

The results of insonating the nylon/cotton and polyester/cotton fabrics at different frequencies and power levels during the finishing process are summarized in Tables III and IV respectively.

Both types of fabric display similar results for each of the treatment cycles examined. In general, a marked increase in weight of finish add-on occurs when the fabric contacts the working edge of the transducer during transport through the fluorochemical bath, as shown by comparing series A, B and C, D as well as I, J. The results also indicate

[b] Ultrasound

TABLE III

Nylon-Cotton (NC-5) Summary of Finishing Results Using Different Treatment Cycles

Sample		Dip &	Treatment	Add-On		U/S Frequency	U/S Power Level	
No.s	Series	Nip	Cycle	Wet Pickup[c] (%)	Net Gain[d] (%)	(kHz)	In (W)	Net (W)
1, 2, 3,	A	2	Tx-Remote	96.59	2.98	-	-	-
4, 5, 6,	B	2	Tx-Contact	109.38	4.92	-	-	-
7, 8, 9,	C	1	U/S-Tx-Remote	104.26	3.68	22.80	200	115
10, 11, 12	D	1	U/S-Tx-Contact	99.09	4.52	22.80	200	115
13, 14, 15	E	1	Tx-Contact	18.61[e]	2.78	-	-	-
16, 17, 18	F	1	U/S-Tx-Contact	-	2.87	8.69	25	14
19, 20, 21	G	1	U/S-Tx-Contact	-	4.44	8.69	200	110
22	H	1	Extra Control	97.31	2.98	-	-	-
23, 24, 25	I	1	Tx-Remote	104.82	3.13	-	-	-
26, 27, 28	J	1	Tx-Contact	98.29	3.42	-	-	-
29, 30, 31	K	1	U/S-Tx-Remote	101.85	3.45	860	100	50
32, 33, 34	L	1	U/S-Tx-Contact	101.46	4.12	46.60	200	110

c Average of three samples; [(damp weight treated - original weight)/ original weight] x 100

d Average of three samples; [(cured weight treated - original weight)/ original weight] x 100

e Samples in this series were not weighed immediately following passage through the treatment bath and padder. The weighings took place after the samples had air dried for 45 minutes. Sample 22 was run through and check weighed immediately.

TABLE IV

Polyester-Cotton (PC-8): Summary of Finishing Results Using Different Treatment Cycles

Sample		Dip &	Treatment	Add-On		U/S Frequency	U/S Power Level	
Nos.	Series	Nip	Cycle	Wet Pickup[c] (%)	Net Gain[d] (%)	(kHz)	In (W)	Net (W)
1, 2, 3	A	2	Tx-Remote	75.63	1.77	-	-	-
4, 5, 6	B	2	Tx-Contact	88.56	3.41	-	-	-
7, 8, 9	C	1	U/S-Tx-Remote	80.58	2.77	22.80	200	115
10, 11, 12	D	1	U/S-Tx-Contact	75.26	3.88	22.80	200	115
13, 14, 15	E	1	Tx-Contact	21.69[e]	2.11	-	-	-
16, 17, 18	F	1	U/S-Tx-Contact	-	2.20	8.69	24	14
19, 20, 21	G	1	U/S-Tx-Contact	-	3.68	8.69	200	110
22	H	1	Extra Control	79.96	2.18	-	-	-
23, 24, 25	I	1	Tx-Remote	87.74	1.61	-	-	-
26, 27, 28	J	1	Tx-Contact	82.26	2.00	-	-	-
29, 30, 31	K	1	U/S-Tx-Remote	80.39	1.98	860	100	50
32, 33, 34	L	1	U/S-Tx-Contact	80.72	2.59	46.60	200	110

that a single dip/nip treatment using an ultrasound-assisted contact mode at high ultrasound power yields about the same level of finish add-on for the given fluorochemical concentration as a two-dip/two-nip contact mode without ultrasound (series B compared to D, G and L). For the light-weight nylon-cotton fabric, the ultrasonic frequency used with the Tx-contact method over the range examined does not appear to be of prime importance in promoting this effect provided enough power is used (e.g. above 100 watts) to ensure cavitation is occuring. Comparing contact mode tests carried out at 8.69 kHz (series F and G) shows the benefits of using power levels in the cavitation range to promote increased finish add-on. In the case of the polyester/cotton fabric, (Table IV) high power levels and lower frequencies, i.e. 8.69 and 22.8 kHz, used with the contact mode appear to produce greater increases in finish add-on compared to contact runs carried out at 46.6 kHz.

An improvement in finish add-on over the standard treatment method also occurs if ultrasound is used in the remote mode in the finishing bath (compare series A, C and I, K). However, the improvement is not as marked as that which occurs with the ultrasound-assisted contact mode and gives roughly the same level of finish add-on as a single dip/nip treatment using the contact mode without ultrasound (series J and K).

The data pertaining to individual samples is given in appendices A and B for the nylon/cotton and polyester/cotton fabrics, respectively.

Laundering Tests

Fabric samples were selected from each series and subjected to the standard laundering test described previously. Water-repellency, oil-repellency and phosphate-resistance properties of the samples were determined periodically up to a total of 12 launder-ometer washings, at which point all samples exhibited relatively poor liquid repellency.

The laundering test results for the nylon/cotton fabric are shown in Table V. In general, using a contact mode of treatment leads to a slight overall improvement in the laundering durability of the fluorochemical finish as compared to the standard (Tx-Remote) treatment method, especially over the first few washes (series A, B and I, J and E). It should be noted that the standard treatment in these tests involved a two-dip/two-nip procedure (series A) or a 3-minute soak in the treatment bath (series I). All contact-mode experiments except series B were run using a single dip/nip procedure. The treated fabrics improve most notably in their phosphate resistance. Using ultrasound with the contact mode or in the remote mode over the range of frequencies and power levels examined does not lead to further improvement in durability of finish properties to laundering, in spite of the fact that a substantial increase in finish add-on has occurred in some cases (series D, G and L).

TABLE V

Laundering Results For Nylon/Cotton Fabric NC-5

Series	Sample Ident.	Finish Add-On (%)	Treatment Method	Wash Number 0	2	4	6	9	12
				100[g]			0		0
A	3	3.0[f]	Tx-Remote	5/6[h]	4/5	4	4	4	3
				9[i]			3		1
				100			0		0
B	5	5.1[f]	Tx-Contact	6	4/5	4/5	4/5	4/5	4
				9			3		2
				100			0		0
C	8	3.6	Tx-Remote	6	4/5	4/5	4/5	4/5	4
			22.8 kHz	9			3		2
				100			0		0
D	11	4.4	Tx-Contact	5/6	4/5	4/5	4/5	4/5	3/4
			22.8 kHz	9			3		1
				100			50		50
E	14	3.0	Tx-Contact	6	6	5	5	5	4/5
				9			6		3
				100			50		50
F	18	2.8	Tx-Contact	6	6	5	5	5	5
			25W, 8.7kHz	9			6		3
				100			50		50
G	20	4.6	Tx-Contact	6	6	5/6	5/6	5	5
			200W, 8.7 kHz	9			6		3
				100			0		0
I	24	3.3[j]	Tx-Remote	6/7	5/6	4/5	4/5	4/5	4/5
				9			3		0
				100			50		0
J	27	3.3	Tx-Contact	6	5/6	4/5	4/5	4/5	3/4
				9			6		3
				100			0		0
K	30	3.3[j]	Tx-Remote	6	5	4/5	4/5	4/5	4/5
			860 kHz	9			1		0
				100			50		0
L	33	4.4	Tx-Contact	6/7	5/6	4/5	4/5	4/5	3/4
			46.6 kHz	9			6		3

f Two-dip/two-nip treatment. All other samples underwent a single-dip/single-nip treatment.

g Water-repellency rating h) Oil-repellency rating

i Phosphate resistance

j Fabric soaked 3 minutes

TABLE VI

Laundering Results For Polyester/Cotton PC-8

Series	Sample Ident.	Finish Add-on (%)	Treatment Method	Wash Number 0	2	4	6	9	12
				80[g]			0		0
A	2	1.9[f]	Tx-Remote	6/7[h]	5	5/6	4/5	4	0-4
				9[i]			0		0
				100			50		0
B	6	3.3[f]	Tx-Contact	6/7	5	5/6	5	5	4/5
				9			0		0
				90			50		0
C	8	2.7	Tx-Remote	6/7	5	5	4/5	4	0-3/4
			22.8 kHz	9			0		0
				90			0		0
D	11	3.7	Tx-Contact	6/7	5	5	5	5	0-4
			22.8 kHz	9			0		0
				100			50		50
E	14	2.2	Tx-Contact	6/7	6/7	6	5/6	5	5
				9			3		0
				100			0		50
F	16	2.3	Tx-Contact	6/7	6/7	6	5/6	5	5
			25W, 8.7kHz	9			3		0
				100			50		0
G	21	3.8	Tx-Contact	6/7	6	5/6	5/6	4/5	4
			200W, 8.7kHz	9			1		0
				100			50		0
I	23	1.6[j]	Tx-Remote	6/7	6	5/6	5/6	5	4
				9			3		0
				100			50		0
J	26	2.1	Tx-Contact	6/7	6/7	5/6	5/6	3/4	3
				9			6		3
				100			50		0
K	29	1.8[j]	Tx-Remote	6/7	6	5/6	5	5	4/5
			860 kHz	9			3		2
				100			50		0
L	34	2.5	Tx-Contact	6/7	6	5	3/4	3/4	3/4
			46.6 kHz	9			3		3

The heavy-weight polyester/cotton fabrics show similar laundering results (Table VI). Again, treatment using a contact mode leads to slight improvement in laundering durability of the imparted repellent properties over the first few washes, especially phosphate resistance. The use of ultrasound does not produce any further improvement over the range of conditions examined.

Wearing Test Results

The experimental wearing machine (Figure 7) used in this study subjects fabric samples to several different kinds of wearing action during each cycle and is useful for comparing the durability of finishes on a common substrate under controlled conditions of fabric tension, humidity and temperature.

Table VII shows the wearing results obtained with the fluorochemical-treated nylon/cotton fabric NC-5. In general, the rate at which liquid-repellent properties deteriorate under the given wearing conditions is nearly identical for the four types of treatment cycles used. This indicates that all treatment methods involving either the single-dip/single-nip or two-dip/two-nip procedure have provided sufficient fluorochemical finish to the fabric to withstand the wearing action of the machine. However, in the case of the polyester/cotton fabric, the data shown in Table VIII indicates that a single-dip/single-nip treatment involving contact between the transducer and fabric (series E and J) leads to slightly improved fabric oil repellency and phosphate resistance on prolonged wearing compared to the standard two-dip/two-nip treatment (series A). No further improvement is gained by using a two-dip/two-nip contact treatment (series B), soaking the fabric for 3 minutes in the treatment bath (series I) or using ultrasound in the contact or remote modes. In some cases, the slight improvement in wearing durability occurs with approximately the same amount of finish added-on to the fabric as compared to the standard treatment (series E, I, J and K compared to A); therefore, although the amount of finish added to the fabric is important in determining the overall level of repellent properties, the manner in which finish is applied in the case of heavy-weight fabrics appears to determine to some extent the durability of these properties to wearing. That is, relatively long soaking times in the treatment bath or contact between a moving fabric and a stationary blade during treatment may produce a somewhat more even deposition of finish or increased fiber penetration by finishing solution compared to the standard treatment method when heavy-weight fabrics are involved.

TABLE VII

Wearing Results For Nylon/Cotton Fabric NC-5

Series	Sample Ident.	Finish Add-On (%)	Treatment Method	Wearing Time (hours) 0	4	8	12	16	20	30
				100[g]					70	70
A	3	3.0[f]	Tx-Remote	5/6[h]	5	5	5	4/5	4/5	4/5
				9[i]	8	6	6	6	6	6
				100					70	70
B	5	5.1[f]	Tx-Contact	6	5	5	5	4	4	4
				9	6	6	6	6	6	4
				100					70	70
C	8	3.6	Tx-Remote	6	5/6	5/6	5	4/5	4/5	4/5
			22.8 kHz	9	6	8	6	6	6	6
				100					70	70
D	11	4.4	Tx-Contact	5/6	5	5	5	4/5	4/5	4/5
			22.8 kHz	9	9	8	6	6	6	6
				100						80
E	14	3.0	Tx-Contact	6	5/6	4/5	4/5	4/5	4/5	4.5
				9	8	6	6	6	6	6
				100						80
F	18	2.8	Tx-Contact	6	5/6	4/5	4/5	4/5	4	4
			25W, 8.7 kHz	9	9	6	6	6	6	6
				100						80
G	20	4.6	Tx-Contact	6	5/6	5/6	5/6	5	4/5	4/5
			200W, 8.7 kHz	9	9	9	8	6	6	6
				100					80	
I	24	3.3[j]	Tx-Remote	6	5/6	5/6	5	4/5	4/5	
				9	8	6	6	6	6	
				100					80	
J	27	3.3	Tx-Contact	6	5	4/5	4/5	4/5	4/5	
				9	9	6	6	6	3	
				100					80	
K	30	3.3[j]	Tx-Remote	6	5/6	5/6	5	4/5	4/5	
			860 kHz	9	8	6	6	6	6	
				100					80	
L	33	4.4	Tx-Contact	6/7	5/6	5	5	5	5	
			46.6 kHz	9	9	6	6	6	6	

f Two-dip/two-nip treatment. All other samples underwent a single/dip single-nip treatment.
g Water-repellency rating
h Oil-repellency rating
i Phosphate resistance
j) Sample soaked for 3 minutes

TABLE VIII

Wearing Results For Polyester-Cotton Fabrics PC-8

Series	Sample Ident.	Finish Add-On (%)	Treatment Method	Wearing Time (hours)						
				0	4	8	12	16	20	30
A	2	1.9[f]	Tx-Remote	80[g] 6/7[h] 9[i]	5/6 9	5 8	5 6	4/5 6	70 4/5 6	70 4/5 3
B	6	3.3[f]	Tx-Contact	100 6/7 9	6 9	5/6 9	5/6 8	5/6 8	70 5/6 8	70 5/6 6
C	8	2.7	Tx-Remote 22.8 kHz	90 6/7 9	5/6 9	5/6 9	5/6 6	5 6	70 5 6	70 5 6
D	11	3.7	Tx-Contact 22.8 kHz	90 6/7 9	5/6 9	5/6 9	5/6 8	5/6 8	70 5 8	70 5 6
E	14	2.2	Tx-Contact	100 6/7 9	6 9	6 9	5/6 9	5/6 9	80 5/6 8	
F	16	2.3	Tx-Contact 25w, 8.7 kHz	100 6/7 9	6/7 9	6 9	5/6 9	5/6 9	80 5/6 8	
G	21	3.8	Tx-Contact 200W, 8.7 kHz	100 6/7 9	6/7 9	6 9	5/6 9	5/6 9	80 5/6 9	
I	23	1.6[j]	Tx-Remote	100 6/7 9	6/7 9	6 9	6 9	5/6 9	80 5/6 8	
J	26	2.1	Tx-Contact	100 6/7 9	6 9	5/6 9	5/6 9	5/6 8	80 5/6 8	
K	29	1.8[j]	Tx-Remote 860 kHz	100 6/7 9	6/7 9	6 9	6 9	5/6 9	80 5/6 8	
L	34	2.5	Tx-Contact 46.6 kHz	100 6/7 9	6 9	5/6 9	5/6 9	5/6 8	80 5/6 8	

Physical Test Measurements

As reported previously (4), insonation of different fabrics at low power levels in the frequency range 50-55 kHz did not significantly affect the strength of the fabrics. In the present study, the tearing strength and breaking strength of selected fabrics subjected to the different treatment cycles were determined according to standard methods. The data shown in Table IX indicates that fabric strength is not affected significantly (greater than 15% change), if at all, by the type of treatment cycle employed. In the case of the nylon/cotton fabric samples, an overall decrease (15-30%) in tearing strength occurs as the result of treating with the fluorochemical finish; a similar result does not occur for the heavy-weight polyester-cotton fabric.

EXPERIMENTS INVOLVING MICROWAVE DRYING

Samples of nylon/cotton and polyester/cotton which had been treated with fluorochemical using the different treatment cycles were dried with 2450 MHz microwaves at a power level of 1500 watts. In most cases, microwave drying was followed by air drying and curing the treated fabrics at 170°C for 2 minutes to insolubilize the fluorochemical polymer.

A summary of the various experiments which were carried out is given in Table X. All of these experiments involved a single-dip/single-nip procedure using the Tx-Contact mode, as described previously. In two cases (NC-5 samples 35 and 36), the fabrics were insonated at 22.8 kHz while polyester/cotton sample number 38 was not oven cured in order to examine the possibility of cross-linking the fluorochemical compound under the influence of microwave energy only.

The treated fabrics were subjected to laundering and wearing tests as before to determine the durability of imparted liquid-repellent properties. Laundering and wearing results are recorded in Tables XI and XII, respectively.

From these results it is evident that microwave-dried fabrics require post-treatment curing in order to fully develop the imparted liquid-repellent properties. This is especially true for the heavier polyester/cotton fabric: the liquid-repellency of sample 38 which was not cured is relatively poor initially and the imparted properties are not as durable to wearing compared to other samples. The phosphate resistance of the uncured polyester/cotton sample unexpectedly improves over the first few hours of wearing and then deteriorates slowly. This unusual result indicates that the finish is not evenly distributed throughout the fabric structure and is not concentrated at the surface of the fabric. A possible explanation for this result is given in the

TABLE IX

Strength Tests of Samples Subjected to Different Treatment Cycles

Sample Ident.	Treatment Cycle	Tearing Strength[k,l] warp (kg)	Tearing Strength[k,l] weft (kg)	Breaking Strength[k,m] warp (kg)	Breaking Strength[k,m] weft (kg)
Nylon/Cotton NC-5					
Control[n]	-	4.82	4.70	64.1	45.0
2	Tx-Remote	3.82	3.60	61.4	48.5
12	Tx-Contact (22 kHz)	3.73	3.64	63.2	49.1
13	Tx-Contact	3.64	3.78	62.7	45.9
16	Tx-Contact (8.7kHz, 25W)	3.73	3.68	59.9	47.7
19	Tx-Contact(8.7kHz, 200W)	3.91	3.55	60.4	46.3
23	Tx-Remote	3.68	3.60	63.1	47.7
28	Tx-Contact	3.86	3.55	60.8	44.9
29	Tx-Remote (860 kHz)	3.73	3.73	62.2	46.3
32	Tx-Contact (46.6 kHz)	3.78	3.37	60.8	48.6
Polyester/Cotton PC-8					
Control[n]	-	4.54	4.40	91.4	90.7
15	Tx-Remote	4.72	4.49	85.8	84.0
17	Tx-Contact(8.7kHz, 25W)	4.81	4.72	84.9	89.9
19	Tx-Contact(8.7kHz, 200W)	4.95	4.68	84.0	86.7
25	Tx-Remote	4.95	4.68	92.6	80.8
27	Tx-Contact	4.77	4.72	91.3	85.8
30	Tx-Remote (860 kHz)	4.90	4.90	92.2	90.3
33	Tx-Contact (46.6 kHz)	4.81	4.86	82.1	84.0

k Average of three runs
l Reference 10
m Reference 11
n Untreated

TABLE X

Summary of Conditions Employed During Experiments Involved Microwave Drying

Sample Ident.	U/S	U/S Freq. (kHz)	M. Wave Passes No.	M. Wave Power (Watts)	Air Dried	Oven Cured	Initial Dry Wt. (g)	After Air Dry or M.W. (g)	After Oven Cure (g)	Air or M.W. Dry Pickup (g)	Air or M.W. Dry Pickup (%)	Final Gain (g)	Final Gain (%)
Nylon/Cotton NC-5													
35	Yes	22.8	1	1500	Yes	Yes	113.5	121.3	118.1	7.8	6.9	4.6	4.1
36	Yes	22.8	2	1500	Yes	Yes	107.8	115.0	111.7	7.2	6.7	3.7	3.6
37	No	-	1	1500	Yes	Yes	116.3	122.3	118.7	6.0	5.2	2.3	2.0
38	No	-	4	1500	No	Yes	106.2	111.7	108.5	5.5	5.2	2.3	2.2
39	No	-	-	-	Yes	Yes	47.7	50.2	48.5	2.5	5.2	0.8	1.7
Polyester/Cotton PC-8													
35	No	-	2	1500	Yes	Yes	143.3	149.3	145.1	6.0	4.2	1.8	1.3
36	No	-	4	1500	Yes	Yes	138.3	143.9	139.7	5.6	4.1	1.4	1.0
37	No	-	6	1500	No	Yes	140.3	145.9	142.1	5.6	4.0	1.8	1.3
38	No	-	6	1500	No	No	140.6	145.8	-	5.2	3.7	-	-
39	No	-	-	-	Yes	Yes	-	123.8	120.6	-	-	-	-

TABLE XI

Laundering Results of Fabrics Subjected to Microwave Drying

Sample Ident.	Repellency Rating/No. of Washes						
	0	1	2	3	4	5	6
Nylon-Cotton NC-5							
35	100 6 9	6	5/6	5/6	5	5	50 4/5 5
36	100 5/6 9	5/6	5/6	5/6	5/6	5	50 4/5
37	100 5/6 9	5	4/5	4/5	4/5	4	0 4 0
38	80 5/6 7	5/6	5	5	4/5	4	0-50 4 0
39	100 5/6 9	4	4	4	4	4	0-50 4 3
Polyester/Cotton PC-8							
35	100 6/7 9	6/7	5/6	5	5	4/5	50 4/5 4
36	100 6/7 9	6	5/6	5/6	5/6	5	50 5 6
37	100 6/7 9	5/6	5/6	5	5	5	50 4/5 6
38	80 5/6 4	5/6	5/6	5/6	5/6	5	50 4/5 0
39	100 6/7 9	6	6	5/6	5/6	5/6	50 4/5 4

TABLE XII

Wearing Results for Fabrics Subjected to Microwave Drying

Sample Ident.	Repellency Ratings Machine Wearing Time (hours) 0	4	8	12	16	20
Nylon/Cotton NC-5						
35	100 6 9	 5/6 6	 5 6	 4/5 6	 4/5 6	80 4/5 6
36	100 5/6 9	 5/6 6	 5 6	 4/5 6	 4/5 6	80 4/5 6
37	80 5/6 9	 4/5 6	 4/5 5	 4 5	 4 5	80 4 3
38	80 5/6 7	 3/4 1	 4/5 0	 4 0	 4 0	80 4 0
39	100 5/6 9	 5 3	 4/5 1	 4 1	 4 1	80 4 1
Polyester/Cotton PC-8						
35	100 6/7 9	 6/7 9	 5/6 9	 5/6 9	 5/6 9	80 5/6 6
36	100 6/7 9	 6/7 9	 5/6 9	 5/6 9	 5/6 9	80 5/6 6
37	100 6/7 9	 6/7 9	 5/6 9	 5/6 9	 5/6 9	80 5/6 6
38	80 5/6 4	 5/6 9	 5/6 6	 5 6	 5 6	80 5 4
39	100 6/7 9	 6/7 9	 6 9	 5/6 9	 5/6 9	80 5/6 6

discussion section which follows. In general, the remaining polyester/cotton samples which underwent microwave drying and a post-treatment cure show similar liquid repellent properties and durability of these properties to laundering and wearing as other samples treated under similar conditions without microwave drying (e.g. sample 37 and samples 14 and 26, Tables VI and VII). Again this indicates that, in the case of the heavy fabrics, post-treatment curing plays a much more important role in the development of desirable liquid repellent properties compared to microwave drying.

The liquid repellency and durability of repellent properties to laundering and wearing of nylon/cotton fabric samples insonated at 22.8 kHz (samples 35 and 36) are comparable to samples previously insonated under the same treatment conditions (sample 11, series D, Tables V and VII). A slight improvement in laundering durability shown by samples 35 and 36 may be attributed to microwave drying, but the contribution of other factors such as differences in finishing bath age, etc. cannot be excluded. It should be noted that the liquid-repellency durability of the control sample in the nylon/cotton series (sample 39) is not as good as samples run previously using the same treatment method (sample 14, series E, Tables V and VII), especially in terms of phosphate resistance. This control sample was run after all other tests had been completed and the deterioration in liquid repellency durability may be due to the introduction of a deleterious factor into the treatment cycle for this fabric.

Using extensive microwave drying without subsequently air drying the nylon/cotton fabric (sample 38) produces unusual wearing results, somewhat similar to those noted for the uncured, microwave-dried polyester/cotton fabric (sample 38). For the lighter fabric, wearing produces an improvement in oil repellency after an initial decrease followed eventually by a slow loss of oil repellency on prolonged wearing. Again, this may be due to uneven deposition of finish material in the fabric structure and lack of concentration of finish at the fabric surface (see Discussion).

DISCUSSION

A sonic or ultrasonic wave of sufficient amplitude produces cavitation, the formation and rapid collapse of small bubbles or cavities, when propagated through a liquid phase such as a fluorochemical treatment solution. These cavitation bubbles take several cycles to grow to what may be called resonant size, at which point they implode violently, producing large, localized pressure changes in the liquid phase.

In the present study, insonation of fabrics during the treatment process generally produced an increase in finish add-on compared to cases where no ultrasound was employed. Provided power levels sufficient to produce cavitation in the treating solution were employed, the increased add-on did not appear to be a function of ultrasonic frequency over the range of frequencies examined. The importance of cavitation in promoting finish add-on is indicated by those insonation experiments carried out at 860 kHz or 8.69 kHz at 25 watts where, in the absence of cavitation, the fabrics received much less finish.

More important, however, is the fact that, for a given treatment bath concentration, a method utilizing contact between the moving fabric and a stationary edge in the treating solution can produce as much or more finish add-on in a single pass as the standard two-dip/two-nip method. Such a method has definite economic advantages in cases where fabrics require treatment with expensive fluorochemical finishes. That is, with the contact method of treatment:

i) A less-concentrated fluorochemical treating solution may be employed to yield a finished fabric which has the desired level of liquid repellent properties, and

ii) A savings in treatment time is gained through using a single-dip/single-nip contact method to provide the same amount of add-on as a two-dip/two-nip treatment.

The beneficial action of the contact method probably arises from localized pressures produced during contact between the stationary edge and moving fabric which forces finishing solution into the fabric structure. Padding equipment for finishing usually incorporates rounded rollers to guide the fabric through the treating solution. With appropriate design to minimize fabric damage during high-speed finishings, it may be possible to use stationary edges (which also act as guides) in place of rollers to provide a contact treatment method.

In all cases (including contact and remote treatment methods), insonation of fabrics at high power levels further increases the amount of finish which can be added on in a single pass. Under the given conditions, there appears to be no advantage in using ultrasound in conjunction with the contact method since the latter method promotes sufficient finish add-on to achieve desired levels of liquid repellency in all cases. However, for fabrics which are difficult to wet with aqueous fluorochemical solution, or for heavier fabrics where increased penetration of finish into the fabric structure is desired, the use of an ultrasound-assisted contact method could be beneficial. For example, it has been shown (4) that fabrics which are difficult to wet under normal circumstances become more readily wetted in the presence of ultrasound. Thus, although not examined in this study, the use of ultrasound may permit higher finishing speeds to be

employed compared to conventional techniques in which effective finishing is limited by the fabric's inability to become thoroughly wetted during high-speed passage through the treating bath.

Prior work has shown that ultrasound may be employed during treatment processes to improve, for example, the dyeability of fibers (12) and the washfastness of crease-resistant finishes (13) and to increase the tanning rate of leather (14, 15). In the present study, in spite of increased finish add-on, the laundering and wearing durability of insonated fabrics was not significantly improved compared to those fabrics treated in the usual fashion or using a contact method. The minor improvement in the wearing durability of the heavier polyester/cotton samples treated in the presence of ultrasound may be due to a more even distribution of finish throughout the fabric but this cannot be stated with certainty at present. Judging from the similar initial repellency ratings of the fabrics which had undergone the four different treatment cycles, it appears that sufficient finish has been added on in each case to cover the outer fabric surface evenly, a necessary condition for producing good liquid-repellent properties (16, 17). With evenly applied finish, the level of repellency exhibited by newly treated or laundered fabrics then depends mainly on the chemical nature of the outermost finish layer (2). Since little fluorochemical finish is lost through laundering under the given conditions (18) and all samples have received the same finish, it is not surprising that a similarity in laundering results occurs for all treatment methods.

In the case of wearing, some differences between the treatment methods with respect to durability of the imparted liquid-repellent properties might be expected to occur. This is because the level of repellency exhibited during wearing depends not only on the evenness and chemical nature of the outermost finish layer but also on how evenly the finish is distributed within the fabric structure (i.e. on how thoroughly the fabric fibers are wetted with finishing solution). That is, as wearing physically removes the outermost finish and fiber layers, underlying newly exposed portions of the fabric can still provide a reasonable level of liquid repellency provided sufficient finish has penetrated to the depth of the now-exposed surface. There are some indications with the heavy polyester/cotton fabric that those methods expected to promote increased penetration of finish (contact and/or insonation in the cavitation range) produce slightly improved levels of liquid repellency on wearing. This is especially true in terms of oil repellency and phosphate resistance which generally are more sensitive indicators of change of finish composition or evenness of coating compared to water repellency. However, very long wearing times (i.e. until the fabric loses much of its physical strength) may be required to firmly establish any significant differences between the treatment methods under the conditions employed. The light nylon/cotton fabric samples which appeared to be thoroughly wetted by the finish solution irrespective of the treatment method used showed very similar wearing results in all cases.

Surface heating (drying and curing) is believed to promote migration of finishes and dyes towards the outermost fabric surface where eventually the finish becomes more concentrated relative to the rest of the fabric (5, 19). That is, conventional surface-heating techniques cause water (carrying finish in solution) to move in liquid form from the wet inner portions of the fabric towards the surface where vaporization takes place, leaving solid finish material behind at or near the point of vaporization. This situation may be contrasted to the case where microwave drying causes vaporization of water throughout the fabric due to localized absorption of the microwave energy and its conversion to heat. Thus, solid finish material is left behind, distributed relatively evenly in the fabric.

With respect to fluorochemical-treated fabrics and maximum durability of imparted repellent properties, it would appear that, for laundering durability, the finish should be concentrated at the fabric surface as can be achieved by conventional surface heating (drying and curing) techniques. Provided the finish is properly insolubilized through curing, relatively little finish is lost through several laundering cycles (18) and the liquid repellency exhibited following laundering depends mainly on whether sufficient heat is supplied during the drying cycle to achieve proper packing and orientation of fluorinated groups in the outermost finish layer (2).

The results indicate that limited microwave drying has no beneficial effect on the laundering durability of the fabrics provided the finish has been air dried and cured in the usual manner. In fact, extensive use of microwave drying (4 or more passes in the microwave guide) prior to or without curing produces lower initial repellency ratings, an indication that the finish is not as concentrated at the surface of the fabric as is the case with conventional surface heating. This also indicates that application of microwave energy has been partially successful in insolubilizing the finish throughout the fabric and preventing migration during subsequent drying and curing steps, especially in the case of the light nylon/cotton fabric. The heavy polyester/cotton samples provide results which are somewhat more difficult to interpret. When no post-treatment curing takes place, extensive microwave drying produces lower initial repellency ratings consistent with having less finish concentrated at the fabric surface. However, if the fabric is then cured, repellency ratings comparable to those obtained with conventional drying and curing techniques are obtained. One possible explanation is that, with the heavier fabrics, insolubilization of finish and drying of the substrate are only partially complete (especially deep within the fabric structure) and during subsequent curing, some migration occurs with eventual concentration and insolubilization of finish at the fabric surface.

The wearing results for the microwave-dried fabrics tend to lend some support to the above explanations. For both the nylon/cotton and polyester/cotton samples which had undergone extensive microwave drying

(without curing in the latter case), wearing for a short time produces some improvement in either phosphate resistance or oil repellency followed by loss of repellent properties at a rate comparable to samples treated in the conventional manner. Again, this is consistent with partial insolubilization of finish through extensive microwave drying followed by soluble fractions migrating towards the surface during curing, especially in the case of the polyester/cotton fabric. However, other explanations for the results cannot be excluded at this time. For example, extensive microwave drying may cause chemical degradation of the outermost finish layers as a result of localized, excessive heating, eventually producing lower initial repellency ratings. This damage could be repaired subsequently through "annealing" of the finish during curing (20) or in response to surface heating produced during wearing. Further work will be required to establish the major factors which operate to produce the observed results.

In general, microwave drying during the finishing process under the given conditions did not produce significant improvement, if any, in the wearing durability of imparted repellent properties compared to standard air-drying and curing techniques.

CONCLUSIONS

1. Under the given conditions, application of ultrasound during treatment of fabrics with water-based fluorochemical finishes leads to significant increases in finish add-on compared to standard treatment methods.

2. Power levels sufficient to produce cavitation in the treating solution are necessary to promote maximum finish add-on when ultrasound is used. When cavitation occurs, the amount of finish added-on is not strongly related, if at all, to the ultrasonic frequency used.

3. Contact between the moving fabric and a stationary blade in the treatment bath provides as much or more finish add-on in a single pass as a standard two-dip/two-nip process. When ultrasound is used with the contact method at power levels sufficient to produce cavitation, a substantial further increase in finish add-on occurs.

4. Insonation over the frequency ranges and power levels used does not result in any adverse effects upon fabric strength.

5. The laundering and wearing durability of imparted liquid repellent properties are not improved to any great extent using the contact method or ultrasound-assisted contact method as compared to a standard treatment method. The overall similarity in laundering and wearing results relates to the fact that all treatment methods examined have provided sufficient finish add-on to produce high levels of liquid repellency.

6. Compared to standard air-drying and curing techniques, the use of microwave drying in conjunction with post-treatment curing does not improve the durability of imparted liquid-repellent properties to laundering and wearing when a contact treatment method is used. Fluorochemical-treated fabrics which have undergone extensive microwave drying with a post-treatment cure possess lower initial liquid-repellency ratings and exhibit unusual wearing behavior as far as liquid repellency is concerned.

SUGGESTIONS FOR FURTHER WORK

The practical benefits arising from the ability of a contact method or ultrasound-assisted contact method to produce a marked increase in finish add-on for a given fluorochemical bath concentration can be established through a series of tests as follows:

1. Using less-concentrated fluorochemical treatment solutions than are normally required, a series of fabric finishing runs using the contact method and/or ultrasound-assisted contact method should be carried out to determine the minimum bath concentration required to produce desired levels of liquid repellency and durability of repellency properties to laundering and wearing.

2. Finishing using the contact method or ultrasound-assisted contact method should be carried out at much higher speeds to determine if a reduction in processing time over conventional treatment methods can be achieved while simultaneously yielding fabrics which possess desired levels of liquid repellency.

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REFERENCES

1. J.M. McAndless and B.V. Lacroix, Defence Research Establishment Ottawa, Report No. 679, July 1973.

2. J.M. McAndless and B.V. Lacroix, Defence Research Establishment Ottawa, Report No. 689, February 1974.

3. J.A. Hart and R.P. Fuoco, Defence Research Establishment Ottawa, unpublished data.

4. J.M. McAndless and B.V. Lacroix, Defence Research Establishment Ottawa, Report No. 741, April 1976.

5. J.E. Pendergrass, J.D. Hatcher and D.W. Lyons, J. Microwave Power 7 (3), 207-213 (1972).

6. D.W. Lyons et al, Text. Chem. Colorist. 9 (5), 86-89 (1977).

7. Launder-Ometer, Wash Test 1, CGSB Standard 4.GP-2 Method 19.1 (1967).

8. American Association of Textile Chemists and Colorists, Water Repellency; Spray Test, AATCC 22-1967, AATCC Technical Manual 44, 154-155 (1968).

9. American Association of Textile Chemists and Colorists, Oil Repellency; Hydrocarbon Resistance Test AATCC 118-1966, AATCC Technical Manual 46, 214 (1970).

10. Tear Strength CGSB Standard 4-GP-2, Method 12.1. Single Rip Method (1957).

11. Breaking Load (Strength) Of Fabrics - Strip Method, CGSB Standard 4-GP-2, Method 9.1 (1962).

12. M. Braver, Melliand Textilber 32, 701-707 (1951).

13. N.N. Simkovich and A.A. Yastrebinskii, Tekhnologiya Tekstil moi Promyshlennosti 3 (106), 15-19 (1975). World Textile Abstracts 7 (17), 1324 (1975).

14. R.L. Ernst and F. Gutmann, J. Soc. Leather Tr. Chem. 34, 454-459 (1950).

15. F. Gutmann. J. Brit. Inst. Radio Engrs. 15, 357-364. (1955).

16. W.A. Zisman, Advances in Chemistry Series No. 43; Contact Angle, Wettability, and Adhesion, Ch. 1, Am. Chem. Soc. (Washington) (1964).

17. E.J. Grajeck and W.M. Petersen, Textile Research J. 32, 320-31 (1962).

18. J.M. McAndless and B.V. Lacroix, Defence Research Establishment Ottawa, Report No. 720, May 1975.

19. D.W. Lyons and C.T. Vollers, Textile Research J. 41, 661-668 (1971).

20. M.K. Bernett, "Oligomeric Fluorinated Additives as Surface Modifiers for Solid Polymers," Proceedings of Chemical Institute of Canada Symposium on Polymers at Interfaces; Focus on Adhesion, October 1976.

Appendix A

Ultrasonics Test Results: Nylon/Cotton NC-5 Samples

Sample No.	Initial Dry Wt. (g)	Wt. After Treatment (g)	After Air Dry (g)	After Cure, 2Min. @ 170°C (g)	Final Wt. & % Gain (g) (%)	Wet Pickup (g) (%)	Remarks
1	15.05	29.35	15.65	15.50	0.45/2.99	14.30/95.02	Control, 2-dip/2-nip
2	15.30	29.90	15.85	15.75	1.45/2.94	14.60/95.42	Tx-Remote
3	15.00	29.90	15.65	15.45	0.45/3.00	14.90/99.34	No U/S
4	17.10	35.95	18.15	17.95	0.85/4.97	18.85/110.23	Triplicate Tx-Contact
5	17.10	37.05	18.75	18.60	0.90/5.08	19.35/109.32	Control (no U/S)
6	16.90	35.25	17.70	17.70	0.80/4.73	18.35/108.58	2-dip/2-nip
7	16.65	33.15	17.35	17.20	0.55/3.30	16.50/99.10	Remote
8	16.60	34.10	17.40	17.20	0.60/3.61	17.50/105.42	Ultrasonics (22.8 kHz)
9	15.75	32.80	16.60	16.40	0.65/4.13	17.05/108.25	Samples 1-dip/1-nip
10	17.10	34.25	17.95	17.95	0.85/4.97	17.15/100.29	Contact
11	15.80	31.45	16.70	16.50	0.70/4.43	15.65/99.05	Ultrasonics (22.8 kHz)
12	16.85	33.35	17.75	17.55	0.70/4.15	16.50/97.92	Samples
13	14.10	15.75	14.65	14.50	0.40/2.84	1.65/11.70	Triplicate Control Samples
14	14.75	17.75	15.35	15.20	0.45/3.05	3.00/20.34	No U/S power, but contacting
15	14.30	17.70	14.85	14.65	0.35/2.45	3.40/23.78	transducer blade. Speed, ca. 2.5 cm/sec.
16	14.85	16.15	15.35	15.25	0.40/2.69	1.30/8.75	Moved @ ca. 2.5 cm/sec.
17	14.35	17.50	15.00	14.80	0.45/3.13	3.15/21.95	25 watts of U/S @ 8.69
18	14.35	17.50	14.90	14.75	0.40/2.78	3.15/21.95	kHz, ie., below cavitation level.

Appendix A (Continued)

Sample No.	Initial Dry Wt. (g)	Wt. After Treatment (g)	After Air Dry (g)	After Cure, 2 Min. @ 170°C (g)	Final Wt. & % Gain (g) (%)	Wet pickup (g) (%)	Remarks
19	14.20	16.30	15.05	14.80	0.60/4.22	2.10/14.70	Similar to 16, 17, 18, but run at 200 watts U/S power, ie. in the cavitation range. Damp weights taken 45 min. after treatment.
20	13.15	14.55	13.95	13.75	0.60/4.56	1.40/10.65	
21	14.35	17.25	15.15	15.00	0.65/4.53	2.90/20.21	
22	13.40	26.45	13.90	13.80	0.40/2.98	13.05/97.39	EXTRA CONTROL Sample to determine true wet pickup value.
23	13.80	28.60	14.25	14.25	0.450/3.26	14.80/107.25	Triplicate Control Samples Soaked 3 min. NOT contacting Transducer.
24	13.75	28.10	14.25	14.20	0.450/3.27	14.35/104.36	
25	14.00	28.40	14.40	14.40	0.400/2.86	14.40/102.86	
26	13.80	27.10	14.40	14.30	0.50/3.62	13.30/96.37	Triplicate control samples-Tx-Contact
27	13.55	26.70	14.15	14.00	0.45/3.32	13.15/97.05	
28	13.65	27.50	14.20	14.10	0.45/3.30	13.85/101.46	
29	13.55	27.70	14.10	14.05	0.500/3.69	14.15/104.43	Triplicate Samples, insonated at 860 kHz.
30	13.45	27.05	13.90	13.90	0.540/3.35	13.60/101.11	
31	13.55	27.10	14.05	14.00	0.450/3.32	13.55/100.00	
32	13.80	27.65	14.50	14.40	0.60/4.3	13.86/100.36	Triplicate samples but with ultrasonic power ON @ 46.60 kHz, 200 watts. (otherwise similar to 26, 27, 28).
33	13.65	27.65	14.40	14.25	0.60/4.39	14.00/102.56	
34	13.60	27.40	14.25	14.10	0.50/3.68	13.80/101.47	

Appendix B

Ultrasonics Test Results: Polyester/Cotton PC-8 Samples

Sample No.	Initial Dry Wt. (g)	After Treatment (Damp) (g)	After Air Dry (g)	After Cure, 2 Min. @ 170 °C (g)	Final Wt. & % Gain (g) (%)	Wet Pickup (g) (%)	Remarks
1	24.80	43.60	25.40	25.25	0.45/1.81	18.80/75.81	Control
2	24.20	42.40	24.80	24.65	0.45/1.86	18.20/75.21	Tx-Remote, 2-dip/2-nip
3	24.45	43.00	24.95	24.85	0.40/1.64	18.55/75.87	No U/S
4	25.70	49.10	26.80	26.55	0.85/3.30	23.4/91.05	Tx-Contact
5	25.90	48.75	27.00	26.85	0.95/3.67	22.85/88.22	Control
6	26.15	48.75	27.25	27.00	0.85/3.25	22.60/89.86	Samples, 2-dip/2-nip
7	24.75	44.90	25.60	25.40	0.65/2.63	20.15/81.41	Remote
8	24.25	43.75	25.10	24.90	0.65/2.68	19.50/80.41	Ultrasonics
9	24.90	44.80	25.75	25.65	0.75/3.00	19.90/79.92	Samples
10	26.95	47.10	28.10	28.05	1.10/4.08	20.15/74.77	Contact
11	25.55	44.90	26.70	26.50	0.95/3.72	19.35/75.73	Ultrasonics
12	25.10	45.75	27.30	27.10	1.00/3.83	19.65/75.29	Samples
13	24.20	28.50	24.85	24.65	0.45/1.86	4.30/17.76	Triplicate control samples
14	24.55	31.90	25.30	25.10	0.55/2.24	7.35/29.94	
15	24.75	29.05	25.45	25.30	0.55/2.23	4.30/17.37	
16	24.30	30.05	25.05	24.85	0.55/2.26	5.75/23.66	Moved @ 2.5 cm/sec., 25 Watts
17	24.05	28.60	24.70	24.55	0.50/2.08	4.55/18.92	@ 8.69 kHz, ie. below the
18	24.30	30.85	25.05	24.85	0.55/2.26	6.55/26.95	cavitation level. Damp weight taken 45 min. after treatment.

Appendix B (Continued)

Sample No.	Initial Dry Wt. (g)	After Treatment (Damp) (g)	After Air Dry (g)	After Cure 2 min @ 170°C (g)	Final Wt. & % Gain (g) (%)	Wet Pickup (g) (%)	Remarks
19	26.50	31.85	27.70	27.45	0.95/3.58	5.35/20.19	Similar to 16, 17, & 18,
20	25.80	31.05	26.95	26.75	0.95/3.68	5.25/20.35	but @ 200 Watts power, in
21	25.05	30.20	26.20	26.00	0.95/3.79	5.15/24.56	the cavitation range.
22	25.20	45.35	25.95	25.75	0.55/2.18	20.15/79.96	EXTRA CONTROL sample to check wet pickup results.
23	21.45	39.75	21.95	21.80	0.35/1.63	18.30/85.31	Triplicate Control samples
24	22.05	41.75	22.55	22.40	0.35/1.59	19.70/89.34	Soaked 3 min., NOT
25	21.85	41.20	22.35	22.20	0.35/1.60	19.35/88.56	contacting transducer.
26	21.50	39.25	22.15	21.95	0.45/2.09	17.75/82.56	Triplicate Control samples
27	21.60	39.50	22.20	22.05	0.45/2.08	17.90/82.87	
28	22.00	39.90	22.60	22.40	0.40/1.82	17.90/81.36	
29	21.80	39.30	22.30	22.20	0.40/1.83	17.5/80.27	Triplicate Samples
30	22.10	40.00	22.65	22.50	0.40/1.81	17.9/80.99	Insonated at 860 kHz.
31	21.65	38.95	22.30	22.15	0.50/2.31	17.3/79.91	
32	22.00	39.90	22.80	22.60	0.60/2.73	17.90/81.36	Triplicate samples
33	22.00	39.90	22.80	22.55	0.55/2.50	17.90/81.36	Tx-Contact
34	21.65	38.85	22.40	22.20	0.55/2.54	17.20/79.45	(46.6 kHz @ 200W).

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Two types of military fabric were treated with a liquid-repellent fluorochemical finish in the presence of high-frequency sonic energy (ultrasound) over a range of frequencies and power levels. Fabric properties such as physical strength, liquid repellency, and durability of repellency to laundering and wearing were evaluated following treatment to determine the effects of using ultrasound during the finishing process. (U)

A second series of tests was carried out to determine whether microwave energy could be employed for the purpose of ensuring a more uniform impregnation of the fabrics, as well as to accelerate the drying and curing processes. (U)

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